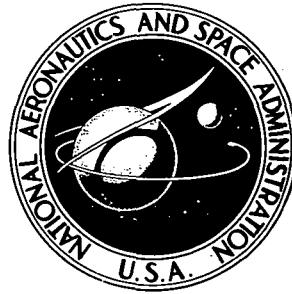


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FRICTION, WEAR, AND TRANSFER OF CARBON
AND GRAPHITE TO COPPER, CHROMIUM,
AND ALUMINUM METAL SURFACES IN VACUUM

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Cleveland, Ohio 44135

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16. Abstract <p>Sliding friction experiments were conducted with amorphous and fully graphitized carbons sliding on copper and on films of chromium and aluminum on copper. Auger emission spectroscopy analysis was used to monitor carbon transfer to the metal surfaces. Friction and wear were also measured. Metal surfaces were examined both in the clean state and with normal oxides present. Results indicate that different metals have an important effect on friction, wear, and transfer characteristics. With amorphous carbon, the least chemically active metal gave the highest wear and amount of carbon transfer. Both forms of carbon gave lower friction and wear and lower transfer rates when in contact with clean, as opposed to oxide-covered, chromium surfaces. With copper, the reverse was true; cleaning was detrimental.</p>			
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SUMMARY

Sliding friction experiments were conducted in this investigation with amorphous and fully graphitized carbons in sliding contact with copper and with films of aluminum and chromium on copper. Friction and wear were measured. In addition, Auger emission spectroscopy analysis was used to monitor the development of carbon transfer films on the metal surfaces. The metal surfaces were examined with the normal surface oxides present and after oxide removal by argon ion sputter cleaning. Friction experiments were conducted with a pin-on-disk specimen configuration.

The results of this study indicate that the chemistry of the metal surface in contact with the carbon is one of the factors that play an important role in friction, wear, and transfer characteristics. With amorphous carbon, the least active metal (copper) gave the highest amount of wear and the most rapidly formed transfer films. In general, the wear of graphitized carbon on all three metals was greater than for amorphous carbon, but the friction was less. With a clean chromium surface, lower friction, lower wear, and slower rate of initial transfer were observed than with oxide-covered chromium. The reverse was true for copper; that is, cleaning was detrimental to these same properties.

INTRODUCTION

Mechanical carbons are widely used in the field of lubrication. They are found in such applications as dynamic mechanical seals (ref. 1), plain journal bearings (ref. 2), and brushes for electrical motors (ref. 3). Further, the graphitic form of carbon is used as a solid-film lubricating material for high-temperature applications (ref. 4) and as an extrusion lubricant (ref. 5).

For most applications, the use of carbons involves the carbon in contact with another material, usually a metal. Experience has indicated that certain metals (e.g., chromium) are better mating surfaces for sliding contact with carbon with respect to friction and wear than are others. The basic reasons for these differences in behavior of carbon with a variation in the metal is not fully understood. Most often, attempts have been made to explain the differences on the basis of mechanical-property (e.g., hardness) differences in the metals.

More recent carbon-to-metal interaction studies have suggested the influence of metal and metal oxide chemistry (refs. 6 and 7). These studies, however, have not attempted to isolate the mechanical properties of the substrate from the chemical role played by the metal surface.

The objective of this investigation was to gain further insight into the influence of metal-surface chemistry (kinetics and thermodynamics of metal interactions with non-metals) on the friction, wear, and transfer characteristics of carbon and metal surfaces in sliding contact. Both amorphous and graphitic carbon were examined in sliding contact with copper and with ion-plated films (200 nm, or 2000 Å) of chromium and aluminum on copper. The metal surfaces were used in the atomically clean state or with the normal oxides present. Auger emission spectroscopy analysis (sensitivity to a depth of 5 atomic layers) was used to monitor changes in surface composition, particularly during initial operation, in dynamic friction and wear experiments.

The carbon specimens used in this study were furnished by the Pure Carbon Company. The physical and mechanical properties of the carbons were supplied by R. Paxton and E. W. Strugala, both of the Pure Carbon Company.

APPARATUS

The experimental apparatus has been described in reference 8 and is depicted in figure 1. The basic elements of the apparatus were the specimens (a 6.35-cm-diameter flat disk and a 0.475-cm-radius rider) mounted in a stainless-steel, bakeable vacuum chamber. The chamber was evacuated from atmospheric pressure by sorption pumps and was then brought to an ultimate pressure of less than 1×10^{-10} torr by an ion pump. The pressure was measured by a cold-cathode trigger-discharge gage in the low-pressure range and by a hot-cathode ionization gage in the millitorr region.

The disk specimen was driven by a magnetic-drive coupling. The rider specimen was supported by an arm that was mounted from a gimbal and sealed to the chamber with a bellows. A linkage at the end of the restraining arm farthest from the rider specimen was connected to a strain-gage assembly that was used to measure frictional force. Load was applied through a deadweight loading system.

An elemental analysis of the wear track on the disk's surface was made before, during, and after sliding by a cylindrical-mirror, electron spectrometer with an integral electron gun. The spectrometer analyzed a 1-millimeter-diameter spot in the wear track of the disk, 153° away from the contact point of the rider. Electrostatic deflection plates in the electron gun permitted the electron beam (and, thus, the analyzed spot) to be moved in and out of the wear track. The Auger spectrum was continuously displayed with a sweep time of 0.1 second on an oscilloscope screen.

In those experiments where carbon-to-metal contact was desired, the disk was cleaned by argon ion bombardment for 1 hour. This was accomplished by applying a 1000-volt negative potential to the specimen in an argon atmosphere at a chamber pressure of 30 to 100 millitorrs. Under these conditions, a glow discharge surrounded the specimen. Although not shown in figure 1, a retractable shield was inserted between the specimens during sputtering to prevent material sputtered from the disk specimen from being deposited on the rider.

MATERIALS

The copper disk specimens used in this investigation were 99.999-percent copper. Ion-plated films, approximately 200 nanometers (2000 \AA) thick, of aluminum and chromium were applied to the copper disks for some experiments. The films were kept very thin to minimize mechanical influences. The aluminum was 99.999-percent aluminum, and the chromium, 99.996 percent.

The amorphous carbon was 93-percent amorphous, with a density of 1.74, Scleroscope hardness of 108, an elastic modulus of 7.3×10^6 kilograms per square meter, and transverse strength of 53 500 kilograms per square meter. The fully (100-percent) graphitized carbon had a density of 1.65, Scleroscope hardness of 50, and elastic modulus of 1.4×10^6 kilograms per square meter, and a transverse strength of 24 900 kilograms per square meter. The carbons were specially prepared, research grades in order to minimize the normal variations in properties common to mechanical carbons.

EXPERIMENTAL PROCEDURE

The disk specimens were polished on metallurgical papers down to 600 grit. They were then polished with 3-micrometer diamond paste and finally polished with 1-micrometer aluminum oxide. They were rinsed with water and ethyl alcohol before they were inserted into the vacuum chamber.

The carbon rider specimens were rinsed with ethyl alcohol before they were inserted into the vacuum chamber. Both the carbon rider and the metal disk were subjected to an overnight bakeout at 250° C in vacuum.

After bakeout of the vacuum system, the pressure in the friction-and-wear apparatus was approximately 10^{-10} torr. At this point, the Auger emission spectrometer was activated. The disk surface was examined with Auger analyses prior to, during, and after sliding contact.

Metal surfaces were examined both with normal oxides present and after argon ion sputter cleaning. A shield was used during sputter cleaning of the disk to avoid the deposition of metallic contaminant on the carbon surface.

The rider was loaded against the disk with a 500-gram load, and sliding was initiated by rotating the disk at a sliding speed of 30 centimeters per minute. During sliding, the surface was continuously monitored with Auger analysis, and friction force was continuously recorded. A total of 100 passes of the rider over the disk were made, and the wear-scar diameter on the carbon was measured to determine wear.

RESULTS AND DISCUSSION

Copper

Both graphitic and amorphous carbon were slid against a copper surface that had the normal residual surface oxide. Auger analysis substantiated the presence of oxygen prior to the initiation of sliding. The friction results obtained in these experiments are presented in figure 2.

The friction coefficient for the amorphous carbon on copper with oxides was very high initially (1.4). After six passes across the surface, the friction decreased markedly, and by the 12th pass, it reached a value of 0.7, where it remained for the balance of the experiment.

The graphitic carbon gave an initial friction coefficient of less than 0.2, and this increased with repeated passes until, at the 15th pass, it reached a value of 0.35, where it remained for the balance of the 100 passes (fig. 2).

The friction coefficient for the graphitized carbon was approximately one-half that of the amorphous carbon after 20 passes. At this point, Auger observations indicated that shear was taking place in the carbon. It is, therefore, worthy to note that the transverse strength of graphitized carbon (from mechanical property data) is one-half that of amorphous carbon.

With both forms of carbon, a film of carbon developed on the copper surface. Transfer of carbon was detected by Auger emission spectroscopy with the very first pass of the

rider across the disk surface. Auger analysis of the sliding contact zone on the disk surface revealed, for the graphitic carbon, a very sharp increase in carbon peak intensity for the first 20 passes. The film at this point was sufficiently thick so that no further increase in carbon peak intensity was observed with additional passes. Auger emission spectroscopy has a limiting detectable film thickness of 5 atomic layers. The amorphous carbon continued to transfer to the copper surface with each successive pass up to 100 passes. A typical transfer film is shown in figure 3. Thus it appears that, with a copper surface, a transfer film develops more rapidly with graphitized carbon. The wear results for both forms of carbon are presented in table I.

The friction coefficient for amorphous carbon sliding on a sputter-cleaned copper disk is also presented in figure 2. The friction coefficient was initially very high (1.6) but decreased with repeated passes to a value of 0.83 at the 20th pass. Beyond 20 passes, the friction did not change. It is of interest to note in figure 2 that both the starting and equilibrium friction values were greater with the sputter-cleaned disk surface than with an oxide-covered surface.

The carbon peak intensity from Auger analysis for graphitized carbon sliding on clean copper is presented in figure 4. Just as with the oxide film present, the intensity for clean copper increases during the first 20 passes, after which no further change in Auger peak intensity for carbon is observed. This indicates that after 20 passes the film is in excess of 5 layers thick.

Unlike the peak intensity of the graphitized carbon, the amorphous-carbon peak intensity continues to increase (fig. 4). At 20 passes, a change in transfer behavior seems to occur with both forms of carbon. With the amorphous film, there seems to be more coverage with each successive pass.

In order to exclude mechanical factors from these studies, hardness measurements were made in and outside the wear track after sliding. The hardness values were within one-half of one point on the Rockwell F scale; this indicates an absence of any extensive hardening in the wear track (approximate hardness of Rockwell F 30).

Surface profilometer traces taken in and outside the wear track indicated an absence of surface roughening of the copper due to sliding.

Sputter cleaning of the copper-disk surface prior to sliding resulted in an increase in both friction and wear for both amorphous and graphitized carbons (see table I). The presence of normal oxide on the surface of copper is, therefore, beneficial to the friction and wear behavior of carbon in contact with copper. It should also be noted that sputtering did not alter surface roughness.

Figure 2 (which gives the friction coefficients for the two forms of carbon sliding on clean and on oxidized copper surfaces) shows that both the form of carbon and the metal surface condition influence friction.

Chromium

Sliding-friction experiments were conducted with amorphous and graphitized carbon sliding against chromium both with its normal oxide present and after sputter cleaning. Friction data are presented in figure 5. Typical Auger traces are presented in figure 6 for graphitized carbon sliding against the oxide-covered chromium surface.

In figure 6(a), Auger emission peaks are seen for the chromium surface before sliding. Auger analysis indicates carbon, oxygen, and chromium peaks. The carbon peak is due to carbon monoxide adsorbed on the surface. Oxygen comes from both the carbon monoxide and the chromium oxide. After 50 sliding passes across the surface, the carbon peak has grown because of the transfer of carbon, while the oxygen and chromium have been attenuated by the overlying carbon film. Upon completion of 100 passes, the only peak detectable is that of carbon. This would indicate a carbon transfer film at least 5 atomic layers thick on the chromium surface.

The carbon peak intensities of graphitic and amorphous carbon transferred to a chromium surface with normal oxide present are compared in figure 7. Unlike the results obtained with copper surfaces, graphitic carbon transferred very rapidly to chromium surfaces. After five passes, no further growth in the graphite peak intensity was observed.

With amorphous carbon, the transfer film developed less rapidly. It took 50 passes of the amorphous-carbon rider across the surface before the carbon peak achieved the intensity level obtained with the graphitic carbon after just five passes. At the 50th pass, both forms of carbon exhibited the same carbon peak intensities. These results indicate that both films are at least 5 atomic layers thick (the limiting thickness detectable with Auger emission spectroscopy).

It is interesting to note in table I and figure 5 that with the normal oxide present, the initial friction coefficient (first pass) of the amorphous carbon is nearly twice the value obtained with electrographitized carbon. After repeated passes and the development of a full transfer film, the difference in values for the two forms of carbon is markedly less.

With residual oxide present on the chromium surface, the initial bonding would be that of carbon to oxygen of the metal oxide. When the chromium surface is clean, the chemical bonding would be carbon to chromium, essentially a metal carbide bond. The bond formed between metal oxides and carbons can be sufficiently strong so as to result in bulk shear in the cohesively weaker of the two materials in contact (ref. 8). The bond strength of carbon to metal oxide may be as high as 12.6×10^6 joules per gram - atom (30 kcal per gram - atom) of carbon. The chromium carbide type of bond for the clean chromium surface may be only 4.2×10^4 to 6.7×10^4 joules per gram - atom (10 to 16 kcal per gram - atom) of carbon (ref. 9). This agrees with the data of figure 5, which indicate that the friction of the graphitic carbon is half that of the amorphous carbon for the clean chromium surface. With the oxide present, there appears to be a strong inter-

action between graphitic carbon and oxygen that results in higher friction than theory would predict.

In figure 8, the carbon Auger peak intensity is plotted for graphitic carbon sliding on a sputter-cleaned chromium surface. Intensity is plotted as a function of the number of passes. The peak intensity continued to increase until a total of 50 passes had been achieved. No further change in Auger peak intensity was then observed.

The Auger carbon peak intensities of figure 8 can be compared with those in figure 7. With a normal oxide present on the chromium surface, the carbon peak growth was complete after just five passes (fig. 7), while 50 passes were required to achieve the same effect (fig. 8) with a sputter-cleaned surface. These results indicate that graphitic carbon transfers more rapidly to an oxide-covered surface.

The strength of the bond formed between carbon and the mating surface appears to influence the rapidity with which a film develops. Thus, the mating surface chemistry exerts an effect beyond the first atomic layer of transferred carbon.

In addition to differences in the rate of carbon transfer for the two surface conditions of the chromium, differences in friction and wear were observed. In general, both friction and wear were lower in the absence of the oxide film than they were with the oxide present (table I). The friction differences existed even after 100 passes, which indicates the important role played by metal surface chemistry. Further, the friction and wear results obtained with a chromium film were the opposite of those obtained with copper. With copper, sputter cleaning resulted in an increase in both friction and wear.

Aluminum

Sliding friction experiments were also conducted on a thin (200 nm, or 2000 Å) film of aluminum present on a copper disk surface. Again, the object was to determine the chemical effects of the aluminum while minimizing the bulk mechanical properties (copper substrate the same).

A typical Auger trace of the aluminum surface prior to sliding is shown in figure 9(a). Peaks for aluminum, carbon, and oxygen are present in the spectrum. The oxygen is a result of both the aluminum oxide present on the surface and the carbon monoxide chemisorbed to the surface.

Some interesting results were obtained when graphitic carbon was rubbed against the oxide-covered aluminum of figure 9(a). The aluminum peak grew in intensity. The increased aluminum peak after two passes is shown in figure 9(b). Note the absence of the oxygen peak. Carbon bonded to the oxygen of the aluminum oxide as it did with chromium oxide. However, unlike the results obtained with the chromium oxide surface, the graphitic carbon sliding on the aluminum oxide surface cleaned the oxide from the surface

and exposed clean aluminum. With repeated passes of the rider over the disk, graphitic carbon began to transfer to the aluminum. The development of a carbon film on the surface can be seen by comparing figures 9(b) and (c).

Even after a total of 100 passes of a graphitic carbon rider over a disk surface and the development of a transfer film of carbon, the metal chemistry influenced the friction results. This is demonstrated by the data of figure 10. After 100 passes and the development of graphite films both on copper (data from fig. 2) and on the aluminum film on copper, differences in friction still existed. The initially high friction coefficient for graphitic carbon sliding on aluminum (first six passes) can not be associated with the shearing in the aluminum film nor with the removal of the oxide film, because even aluminum has a higher shear strength than does the graphitic carbon.

In figure 11, the relative Auger peak intensities for amorphous and graphitized carbon sliding on the aluminum film are presented. With amorphous carbon, the rate of carbon transfer to the aluminum is less than the observed rates of transfer to copper and to chromium. Furthermore, the difference between the buildup rates of amorphous and graphitized carbon is greater than was observed between these two forms of carbon on copper and on chromium. Some of this difference may be due to natural shearing of aluminum by amorphous carbon, since amorphous carbon, in contrast to graphitized carbon, has a higher shear strength than aluminum.

Figure 12 summarizes the Auger cylindrical mirror analysis of the three metal surfaces during sliding contact with amorphous carbon. The greatest rate of carbon transfer occurs with copper, the least chemically active metal; the smallest rate of carbon film transfer occurs with aluminum, the most chemically active metal. This occurs despite the fact that copper forms no stable carbide structure with carbon (ref. 9) and carbon is practically insoluble in copper (ref. 10).

The wear of amorphous carbon correlates with the film-transfer characteristics shown in figure 12. Table I shows that wear is greatest for the carbon in contact with copper and least for contact with aluminum. Thus, for amorphous carbon, the more chemically active the metal surface, the thinner the carbon transfer film and the lower the wear of the carbon.

With the aluminum film, there is a mechanical shear factor which must be considered. The shear strength of aluminum is higher than that of electrographitized carbon but lower than that of amorphous carbon. Thus, even though thin films (200 nm, or 2000 Å) of aluminum were used, shearing in that film when contacted by amorphous carbon could have influenced the observed results.

SUMMARY OF RESULTS

The sliding-friction studies of this investigation with amorphous and graphitized carbon bodies in sliding contact with surfaces of copper, aluminum, and chromium films yielded the following results:

1. The chemical nature of metal surfaces, aside from their differences in mechanical properties, influences the behavior of mechanical carbons with respect to friction, wear, and transfer characteristics.

2. The least amount of wear for amorphous carbon occurred when in sliding contact with the chemically most active and the softest metal, aluminum. Auger analysis indicated that the aluminum surface also had the least amount of transferred carbon.

3. With graphitic carbon, the sputter-cleaned chromium surfaces produced lower friction and wear and less initial transferred film material than did the oxide-covered surfaces. With copper surfaces, sputter cleaning produced greater friction and wear.

4. In general, the wear of graphitic carbon against all three metal surfaces was higher than that of amorphous carbon, while the average friction was less. Furthermore, the graphitized carbon transferred to metals more rapidly than did the amorphous carbon.

5. Even after 100 passes of a carbon rider over a disk and the development of a carbon transfer film, friction results were still influenced by the underlying surface.

In general, the use of carbons in practical applications requires consideration of the metal surface as well as the carbon form. The more active metal will give the thinner transfer films. Graphitic carbon will give lower friction, while the use of amorphous carbon will result in less wear.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, December 18, 1972,
502-01.

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TABLE I. - COEFFICIENT OF FRICTION AND CARBON WEAR
FOR VARIOUS CARBON-METAL COMBINATIONS

Metal surface	Carbon	Surface condition	Friction coefficient		Wear volume per revolution, 10^{-6} cm^3
			First sliding pass	Stabilized average	
Copper	Graphitic	Normal oxide present	0.15	0.31	5.95
		Sputter cleaned	0.61	0.40	8.66
	Amorphous	Normal oxide present	1.2	0.7	2.95
		Sputter cleaned	1.5	0.83	3.80
Chromium	Graphitic	Normal oxide present	0.41	0.60	3.68
		Sputter cleaned	0.42	0.40	2.95
	Amorphous	Normal oxide present	0.80	0.86	2.44
		Sputter cleaned	0.80	0.80	1.47
Aluminum	Graphitic	Normal oxide present	0.80	0.50	8.90
		Sputter cleaned	1.0	0.45	7.95
	Amorphous	Normal oxide present	0.85	0.80	1.15
		Sputter cleaned	0.96	0.80	1.06

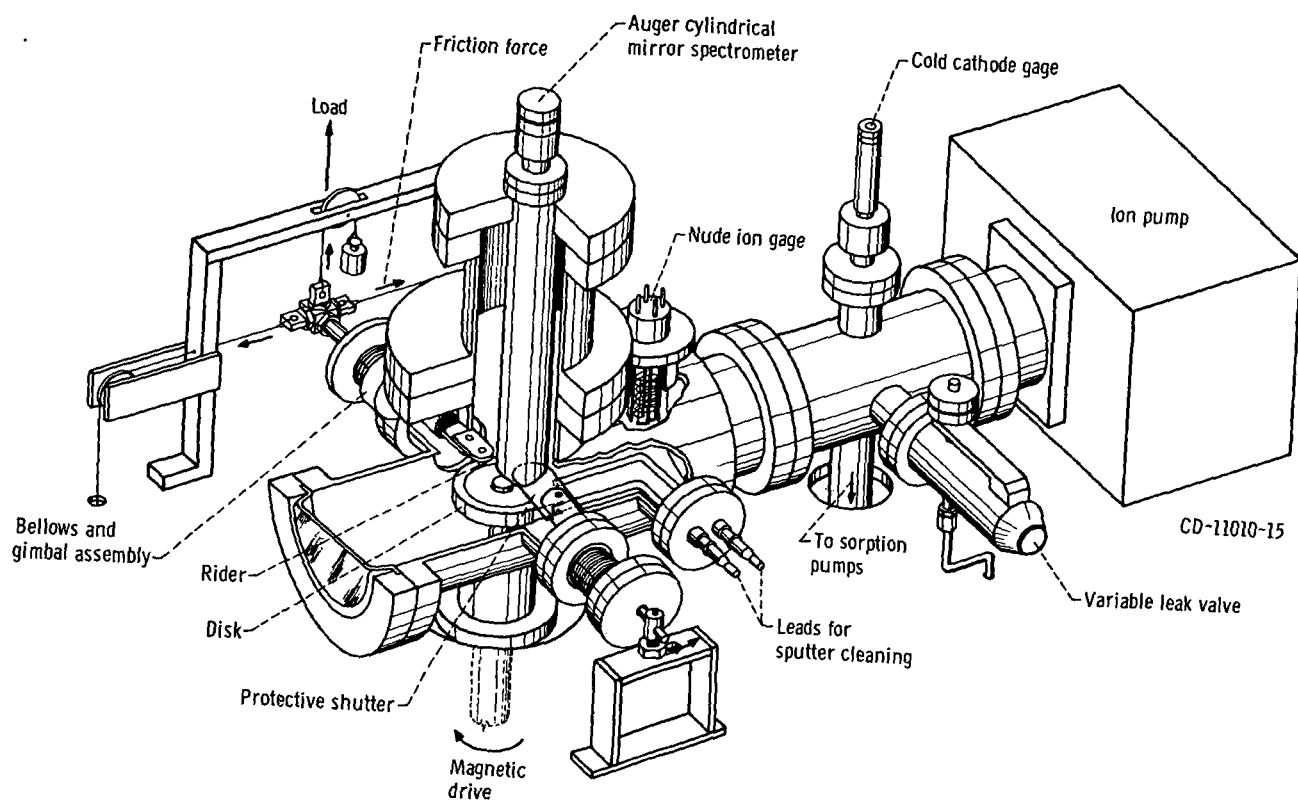


Figure 1. - Friction apparatus with Auger spectrometer.

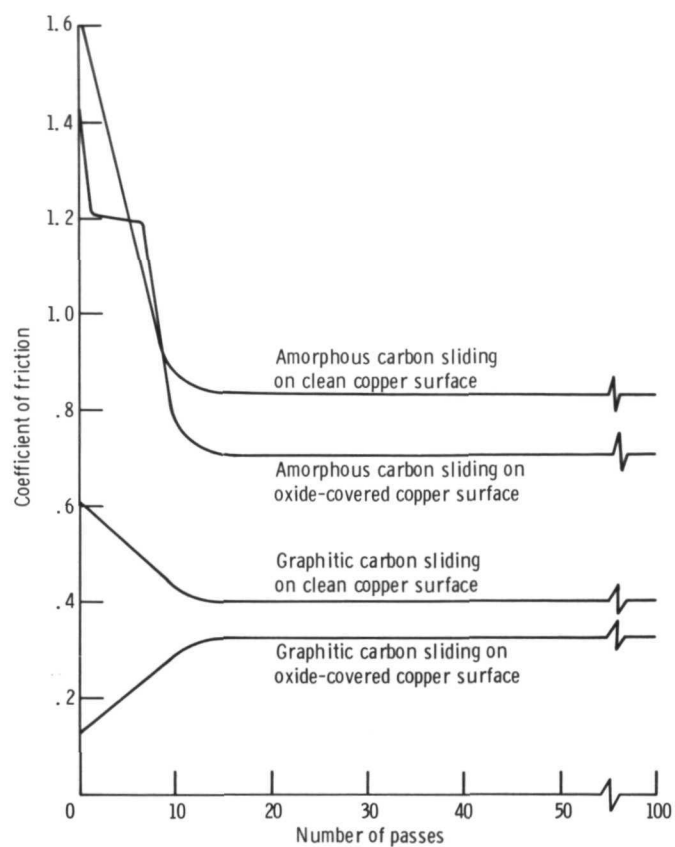
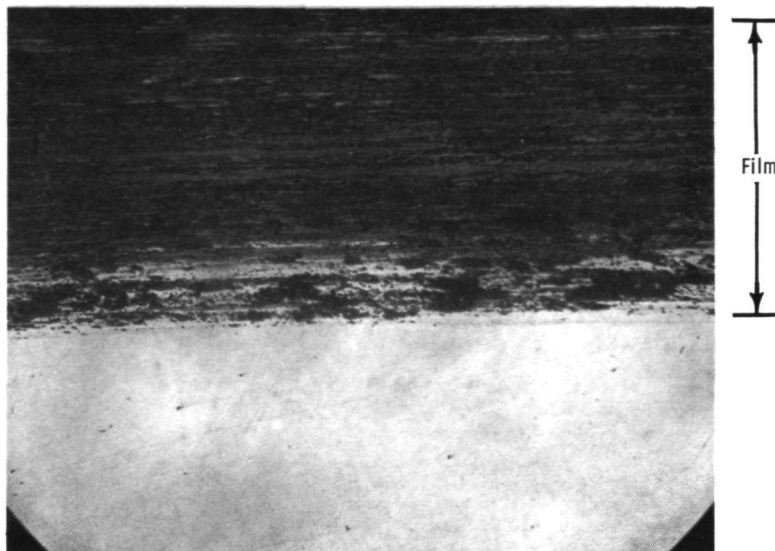
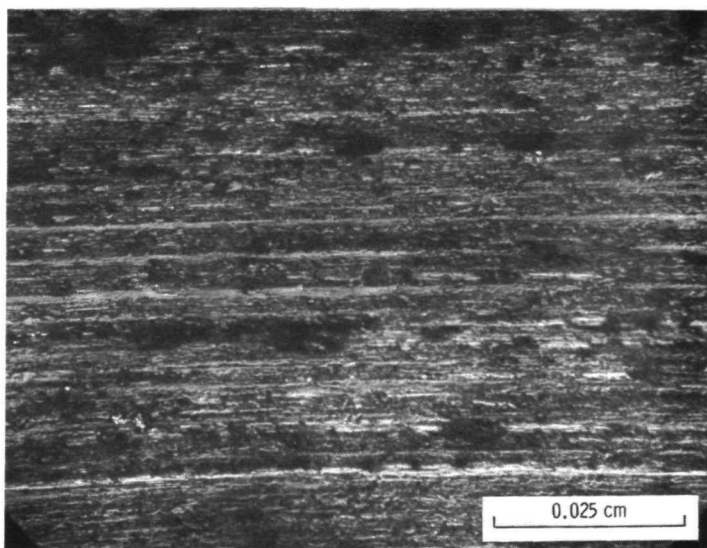


Figure 2. - Comparison of friction coefficients for two forms of carbon sliding on clean and on oxidized copper. Sliding velocity, 30 centimeters per minute; load, 500 grams; ambient temperature, 23° C; ambient pressure, 10^{-10} torr.



(a) Film and disk surface.



(b) Film.

Figure 3. - Photographs of carbon transfer film on sputter-cleaned copper surface. Sliding velocity, 30 centimeters per minute; load, 500 grams; ambient temperature, 23° C; ambient pressure, 10^{-10} torr.

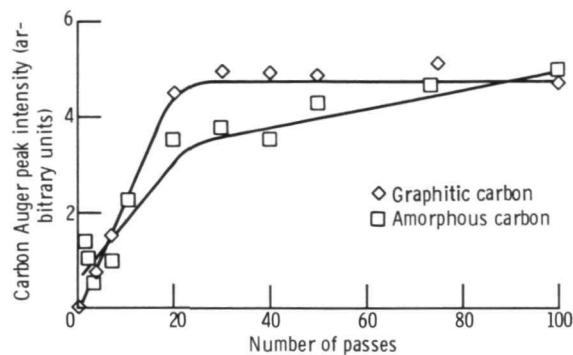


Figure 4. - Carbon peak intensity as a function of the number of sliding passes for graphitic carbon and amorphous carbon sliding on sputter-cleaned copper surfaces. Sliding velocity, 30 centimeters per minute; load, 500 grams; ambient temperature, 23° C; ambient pressure 10⁻¹⁰ torr.

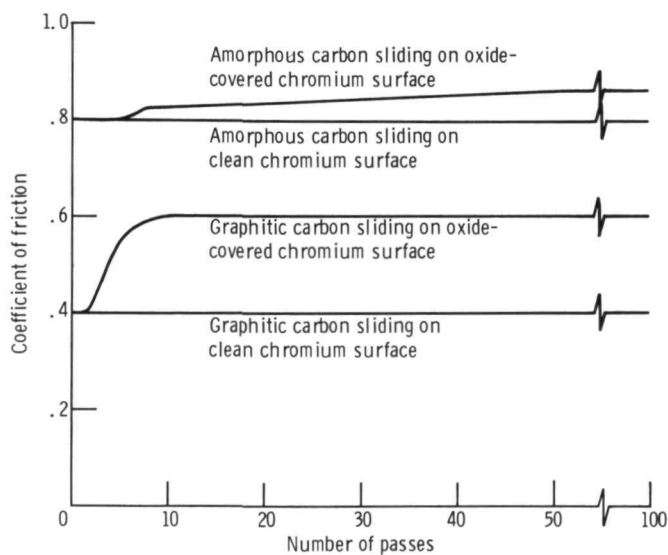
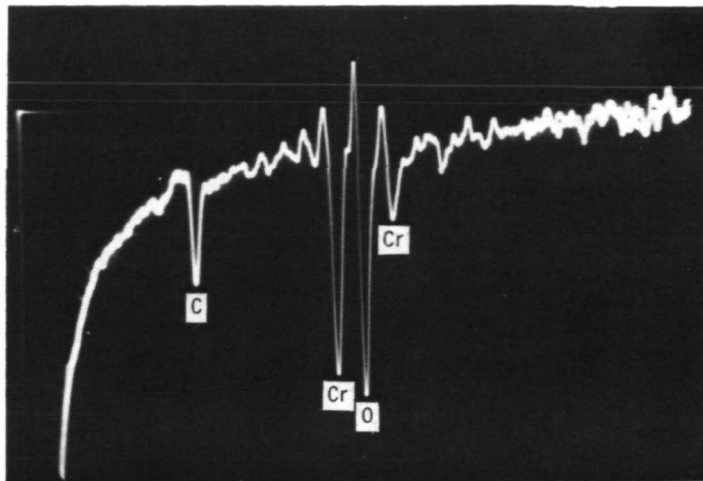
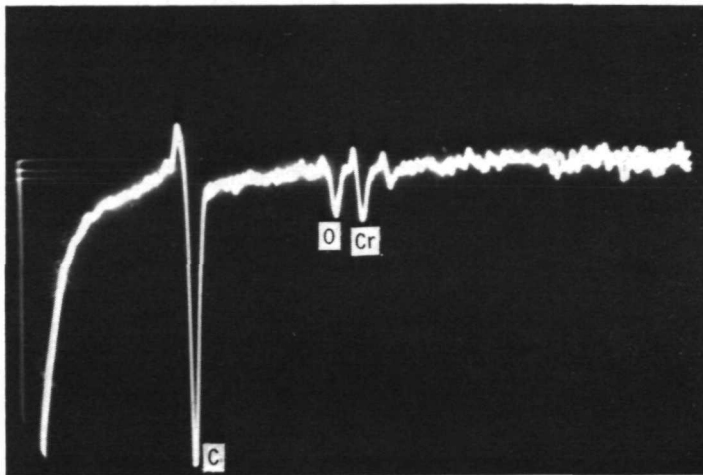


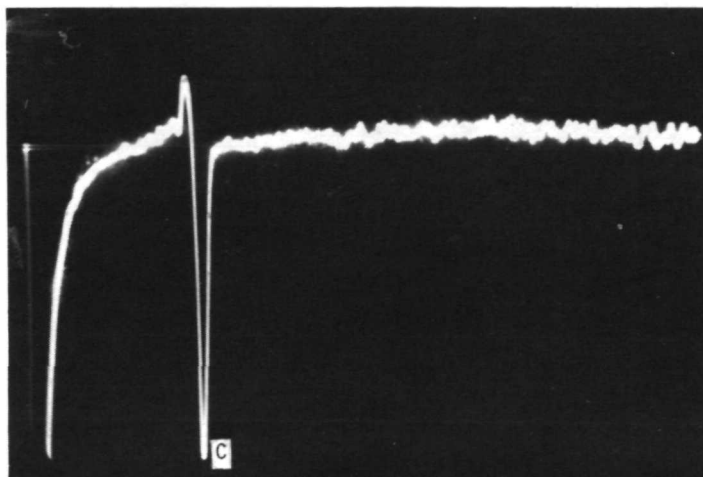
Figure 5. - Coefficients of friction for two forms of carbon sliding on clean and on oxidized chromium. Sliding velocity, 30 centimeters per minute; load, 500 grams; ambient temperature, 23° C; ambient pressure, 10⁻¹⁰ torr.



(a) Before sliding.



(b) After 50 sliding passes.



(c) After 100 sliding passes.

Figure 6. - Photographs of oscilloscope display of oxide-covered chromium surface film and the development of a graphite transfer film.

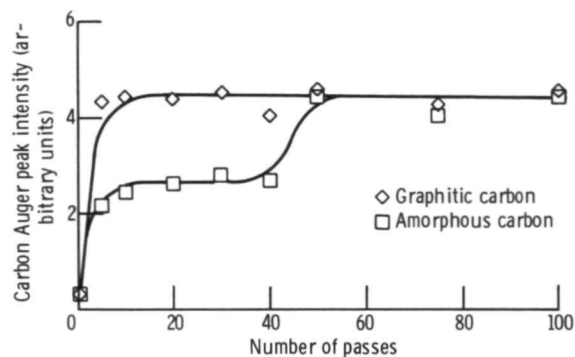


Figure 7. - Carbon Auger peak intensity as a function of the number of sliding passes for graphitic and amorphous carbon sliding on oxide-covered chromium surfaces. Sliding velocity, 30 centimeters per minute; load, 500 grams; ambient temperature, 23° C; ambient pressure, 10^{-10} torr.

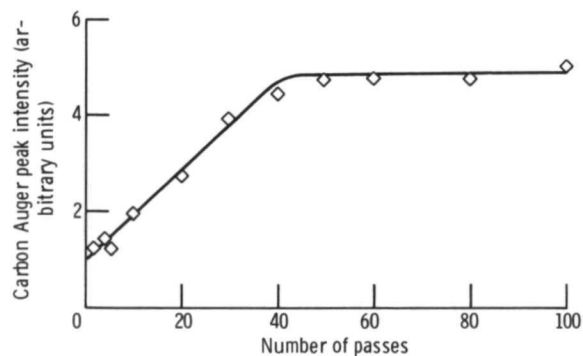
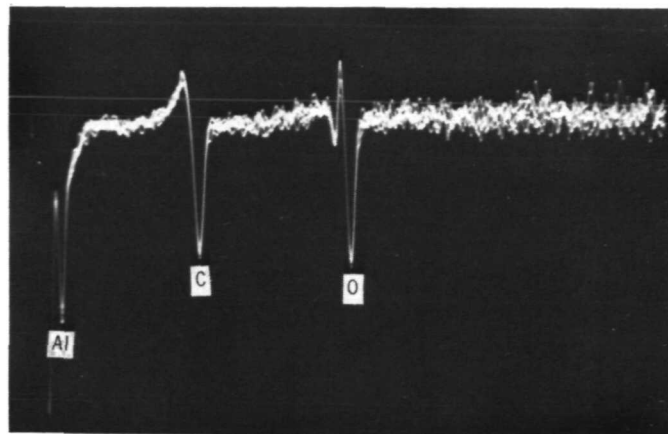
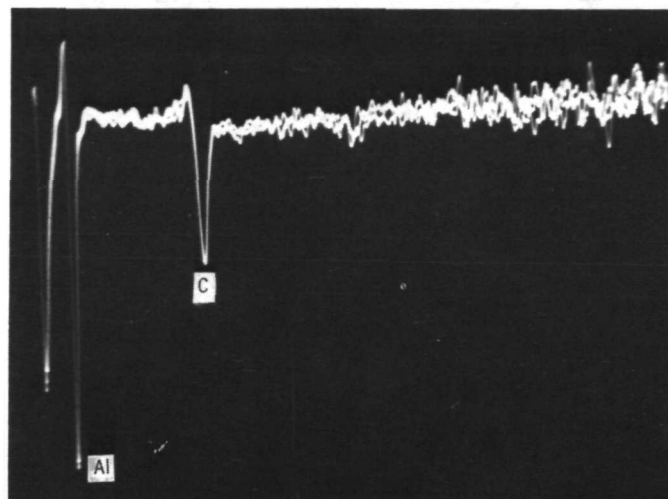


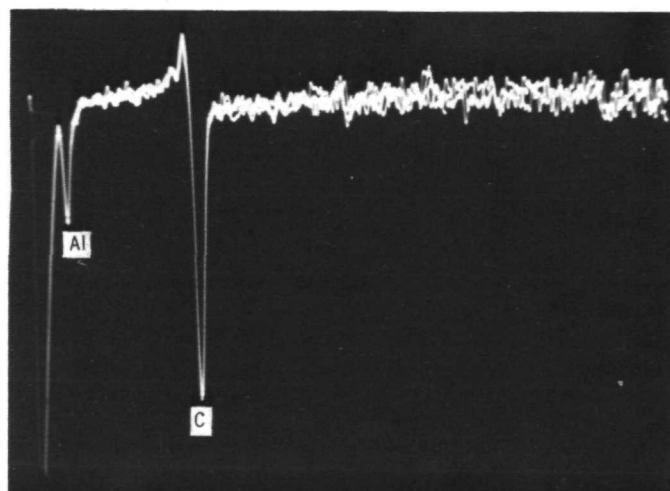
Figure 8. - Carbon peak intensity as a function of the number of sliding passes for graphitic carbon sliding on a sputter-cleaned chromium surface. Sliding velocity, 30 centimeters per minute; load, 500 grams; ambient temperature 23° C; ambient pressure, 10^{-10} torr.



(a) Before sliding.



(b) After 2 sliding passes.



(c) After 100 sliding passes.

Figure 9. - Photographs of oscilloscope traces of an aluminum surface film before and after the transfer of graphitic carbon.

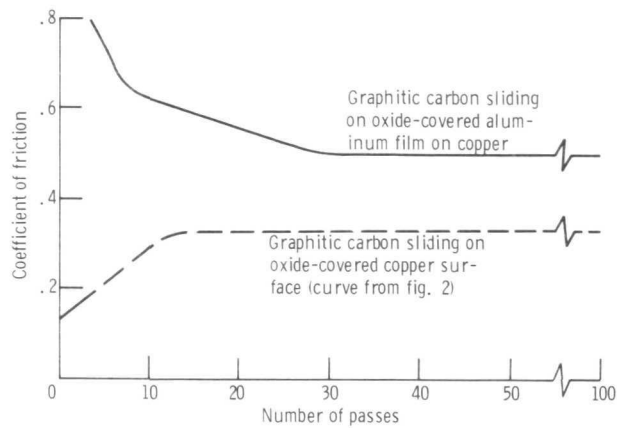


Figure 10. - Coefficients of friction for graphitic carbon sliding on an oxide-covered aluminum film on copper and on an oxide-covered copper surface. Sliding velocity, 30 centimeters per minute; load, 500 grams; ambient temperature, 23° C; ambient pressure, 10⁻¹⁰ torr.

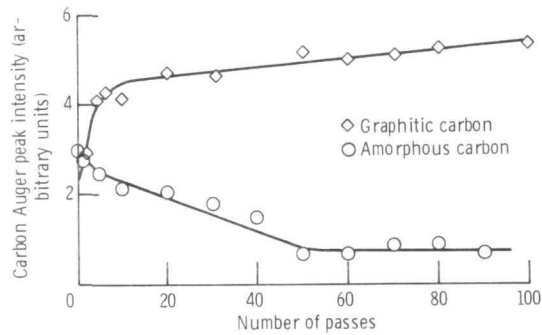


Figure 11. - Carbon Auger peak intensities for graphitic and amorphous carbon sliding on an aluminum surface with normal oxide present. Sliding velocity, 30 centimeters per minute; load, 500 grams; ambient temperature, 23° C; ambient pressure, 10⁻¹⁰ torr.

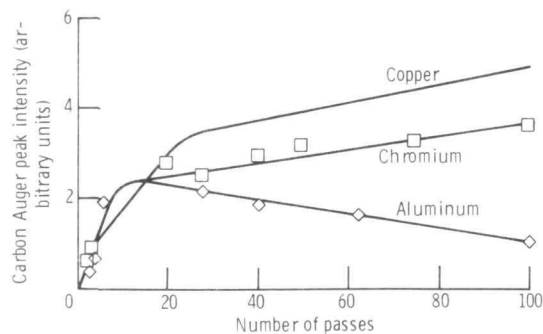


Figure 12. - Carbon Auger peak intensities for amorphous carbon sliding on three sputter-cleaned metal surfaces. Sliding velocity, 30 centimeters per minute; load, 500 grams; ambient temperature, 23° C; ambient pressure, 10⁻¹⁰ torr.



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